
An Overview of the Hydrogen Storage Materials

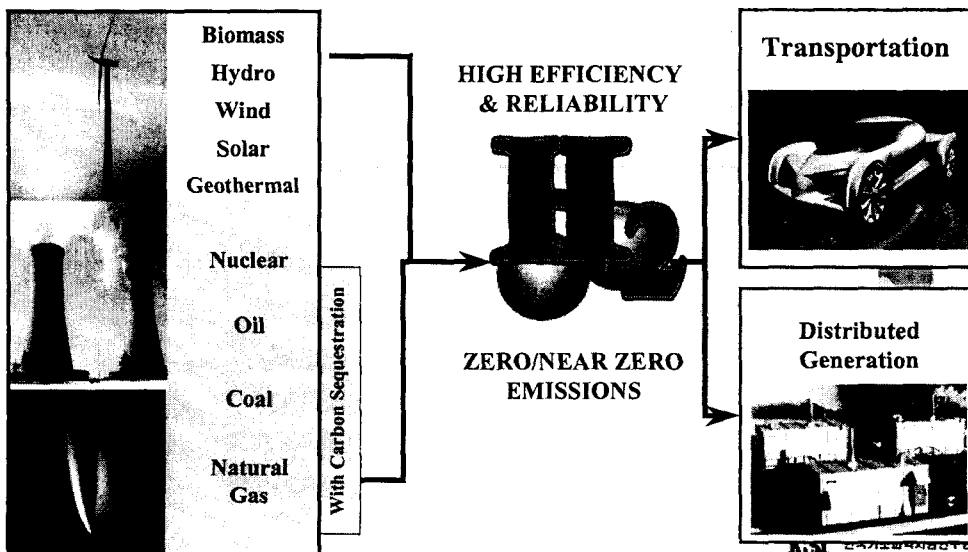
김 해 진 박사
(한국기초과학지원연구원)

An Overview of the Hydrogen Storage Materials

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Frontier Research Laboratory
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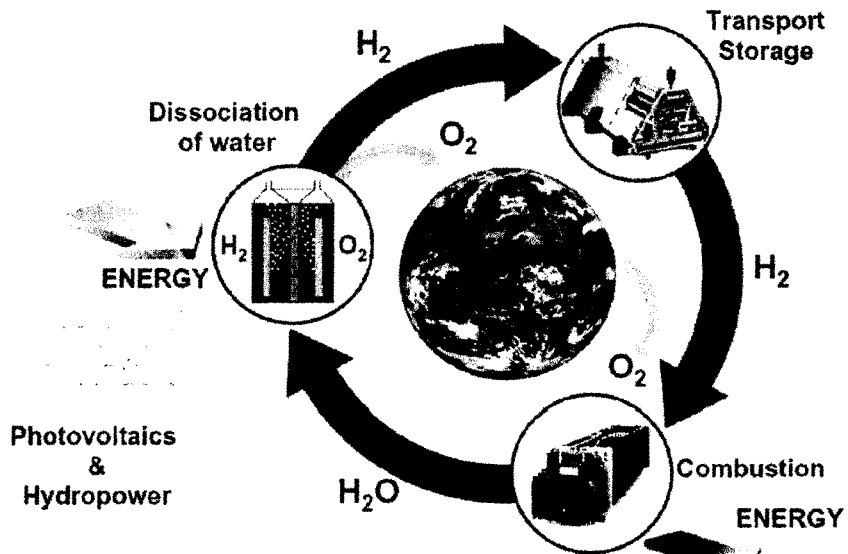


Why Hydrogen? It's abundant, clean, efficient,
and can be derived from diverse domestic resources.



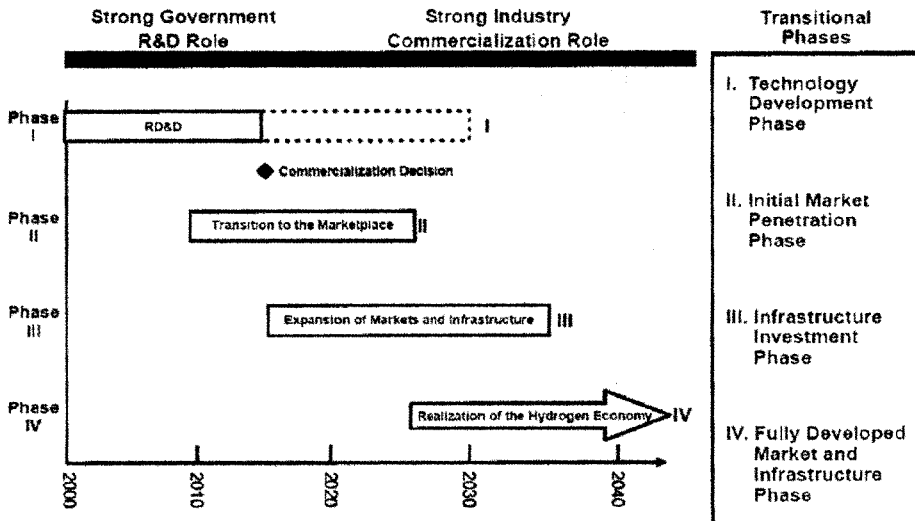
HYDROGEN CYCLE

Sun



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Time for Hydrogen Economy



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DOE Hydrogen Storage R&D Program Approaches

Chemical Storage (2004)

- *NaBH₄ Process Chemistry*
- *Life-Cycle Analyses*
- *Other Hydrides*

Complex Metal Hydrides

- *NaAlH₄ System Integration*
- *Hydride Materials R&D*
- *Kinetics/Mechanistic Studies*

Standard Testing Procedures/Facilities

Advanced Concepts (2004)
- TBD

Carbon

- *Kinetics/Mechanistic Studies*
- *Process R&D*
- *Structure/Property Analyses*

Compressed/Liquid Tanks

- *5,000/10,000 psi Tanks*
- *Semi-Conformal System*
- *Tank Liners/Overwrap Materials*
- *Insulated Pressure Vessels*
- *Unusual Shapes*

From Patrovic & Milliken (2003)

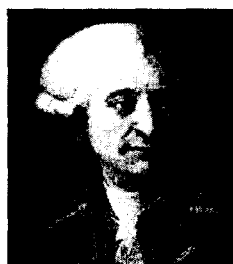


History

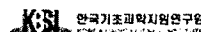
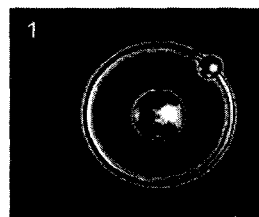
- Discovered about 200 years ago (1766)



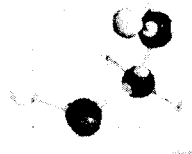
Henry Cavendish (1731-1810)



Antoine Lavoisier (1743-1794)



History



- In 1931, hydrogen was discovered to have isotopes



vs.

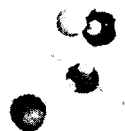


Harold C. Urey (1893-1981)
Nobel Laureate 1934

Frederick Soddy (1877-1956)
Nobel Laureate 1921



Isotopes of Hydrogen



- Three common isotopes:
 - Protium (H)
 - common hydrogen
 - 99.985% abundant
 - Deuterium (D)
 - one neutron
 - 0.015% abundant
 - Tritium (T)
 - two neutrons
 - $1 \times 10^{-15}\%$ abundant



Protium

1 Proton

0 Neutron

Henry Cavendish (1776)



Deuterium

1 Proton

1 Neutron

Harold C. Urey (1931)



Tritium

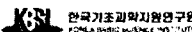
1 Proton

2 Neutrons

Ernest Rutherford (1934)

Unstable with a half-life of 12.43 years

H/D=6000



Two groups of H molecule

$I = 0$, antiparallel nuclear spin

$I = 1$, parallel nuclear spin

Para



Ortho



- Normal Hydrogen is 75% Ortho, 25% Para (at 298 K)
- The melting and boiling points of para hydrogen are ca. 0.1 K lower than those of normal hydrogen.
- At $T=0$ K, ALL the molecules must be in a rotational ground state (in the para form)
- The ortho form cannot be prepared in the pure state

Mass energy densities for various fuels

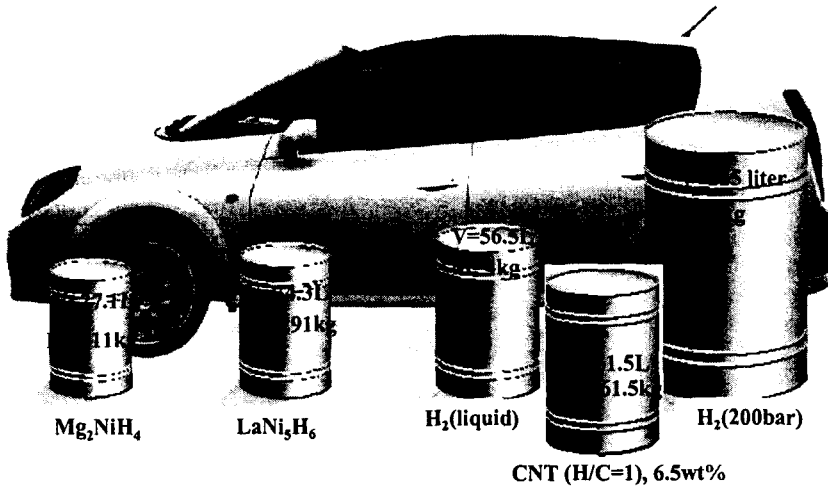
Increasing molecular wt.

Fuel	Hydrogen weight fraction	Ambient state	Mass energy density (MJ/kg)
Hydrogen	1	Gas	120
Methane	0.25	Gas	50 (43) ²
Ethane	0.2	Gas	47.5
Propane	0.18	Gas (liquid) ¹	46.4
Gasoline	0.16	Liquid	44.4
Ethanol	0.13	Liquid	26.8
Methanol	0.12	Liquid	19.9

- (1) A gas at room temperature, but normally stored as a liquid at moderate pressure.
 (2) The larger values are for pure methane. The values in parantheses are for a "typical" Natural Gas.

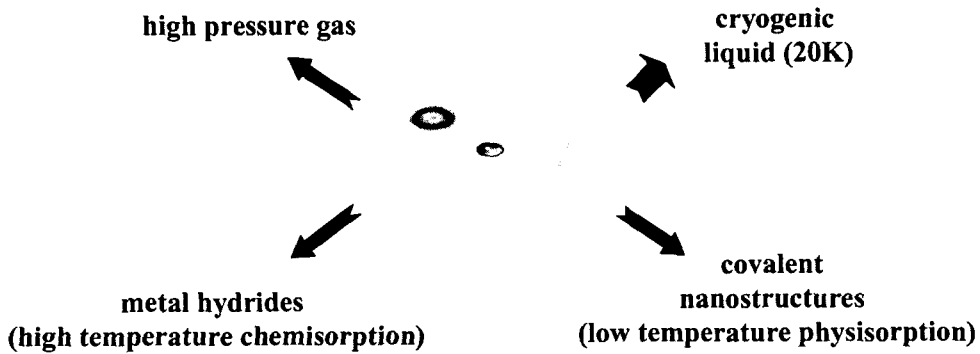
How large of a gas tank do we want?

Volume Comparisons for 4 kg Vehicular H₂ Storage



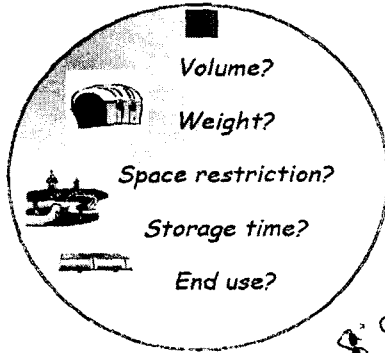
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Hydrogen Storage Options

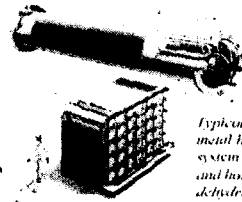


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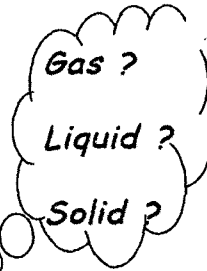
Hydrogen storage Technology



Stationary applications

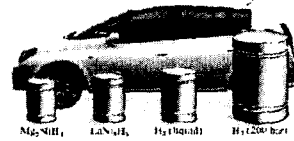


Typical Layout of a metal hydride storage system with heat exchanger and hot water taps for dehydrogenating



Mobile

Volume of 1 kg of hydrogen compacted in different ways, with volume relative to the size of a car

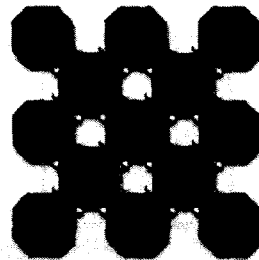


Mobile- bigger challenge than stationary!

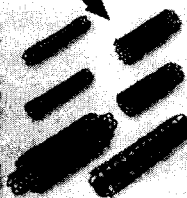
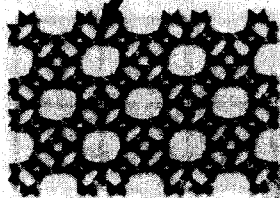
Hydrogen Storage Materials

High H-mass density
High H-volume density
Appropriate p, T stability
Reversible absorption/desorption

metal hydrides
carbon based materials
microporous materials



Metal hydride forming elements
"Rule of 2 Å" for H-H separation

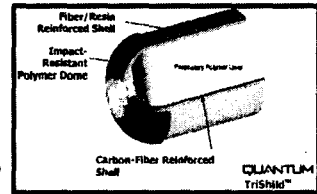
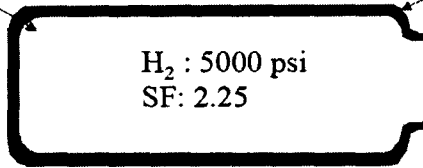


Compressed Hydrogen Storage



Aluminium/Thermoplastic

Glass/Carbon fibre

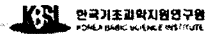


Composite H₂ Cylinder – 12 wt%

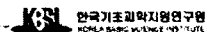
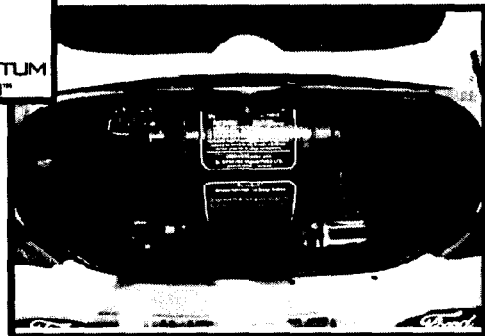
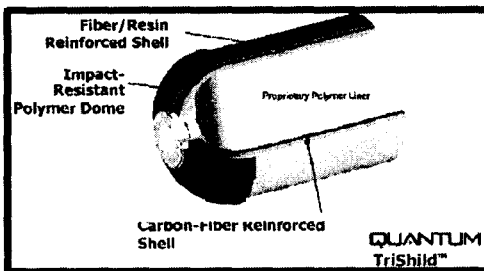
Conformable geometrics

Higher wt% via increased pressure

Heating on filling



Compressed Hydrogen



Liquid Hydrogen Storage

- Liquid Hydrogen Storage
- Cryogenic storage of hydrogen @ -253°C

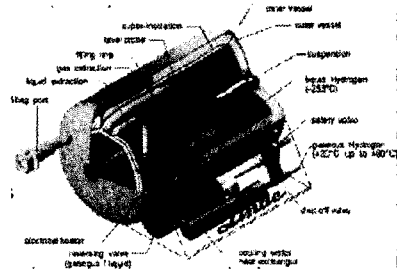
- Advantages

- ✓ Low pressure
- ✓ High storage density

- Disadvantages

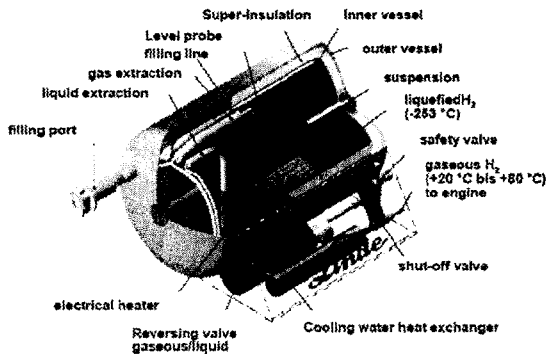
- ✓ Energy required for liquefaction
- ✓ Evaporative losses during fueling
- ✓ Evaporative losses during periods of inactivity, i.e. when parked
- ✓ Consumer Acceptance

- Future developments to improve packaging and reduce evaporative losses

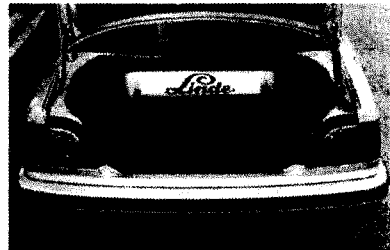
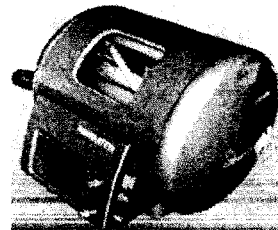


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Liquid Hydrogen Development of Storage Technology

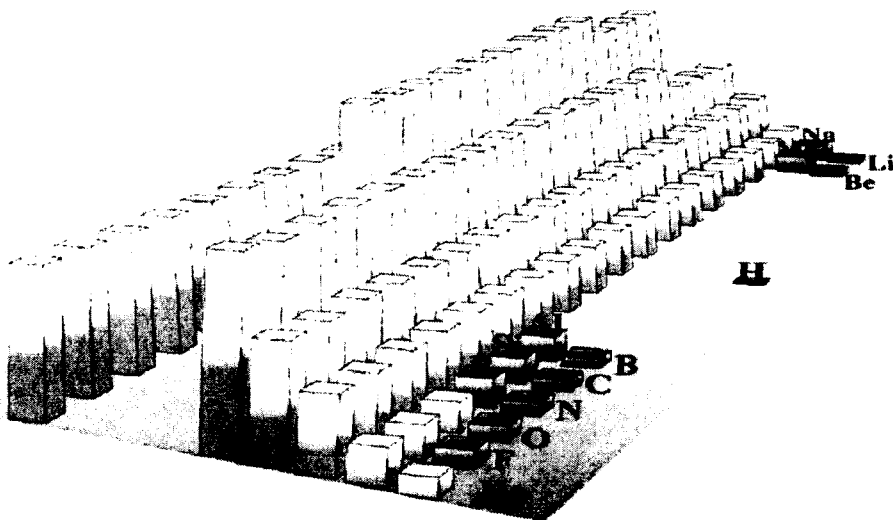


Automotive Design:



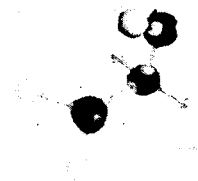
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The Periodic Table of the Chemical Elements
The mass of each element is indicated by elevation above the plane




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Hydrides

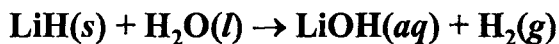


- Binary compounds of hydrogen
 - has an intermediate electronegativity
 - ionic hydrides
 - LiH
 - covalent hydrides
 - HF
 - metallic hydrides
 - NiH₂

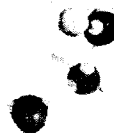
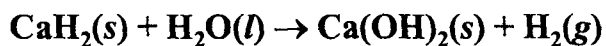
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Ionic Hydrides

- white solids
- metal cation and hydride ion
- very reactive

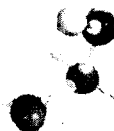


- reducing agents

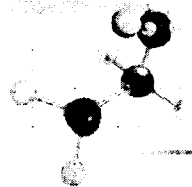


Covalent Hydrides

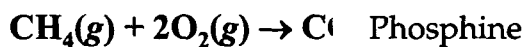
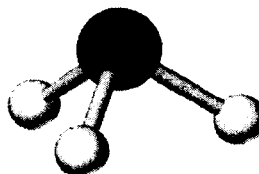
- covalently bonds with all nonmetals and weakly electropositive metals
- gases at room temperature
 - hydrogen can be:
 - nearly neutral
 - substantially positive
 - slightly negative



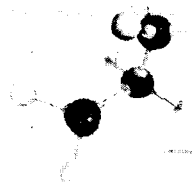
Neutral Covalent Hydrides



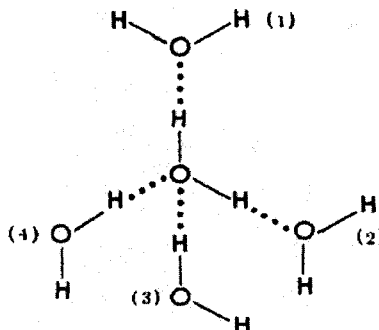
- low polarity
 - only dispersion forces
- Examples:
 - PH_3
 - CH_4
 - Hexene



Positive Covalent Hydrides



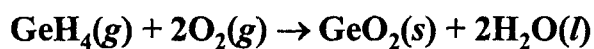
- high melting and boiling points
 - protonic bridging
- Examples:
 - ammonia
 - water
 - hydrogen fluoride



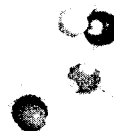
Negative Covalent Hydrides



- Contains hydridic hydrogens
- Very reactive towards oxygen
- Examples:
 - B_2H_6
 - SiH_4
 - GeH_4

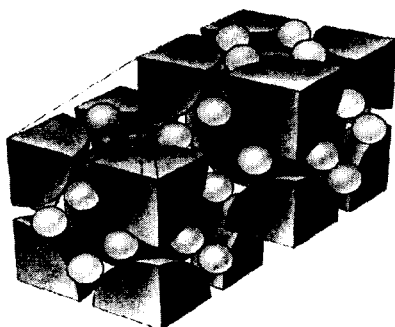


Metallic Hydrides

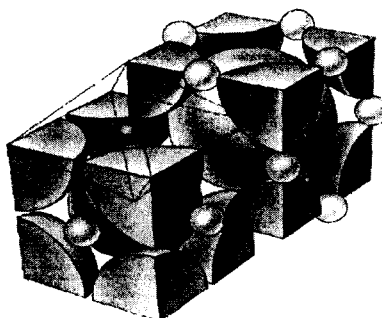


HYDROGEN INTERCALATION IN METALHYDRIDES

HYDROGEN
ON
TETRAHEDRAL SITES



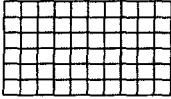
HYDROGEN
ON
OCTAHEDRAL SITES



HYDROGEN ABSORPTION



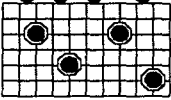
Hydrogen gas



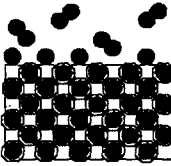
Metal



α -Phase: Solid Solution

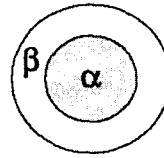


MH_x ($0 < x < 0.1$)
 $H \rightleftharpoons H$, $\Delta VV = k \cdot c_H$

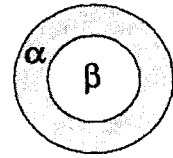


β -Phase: Hydride Phase

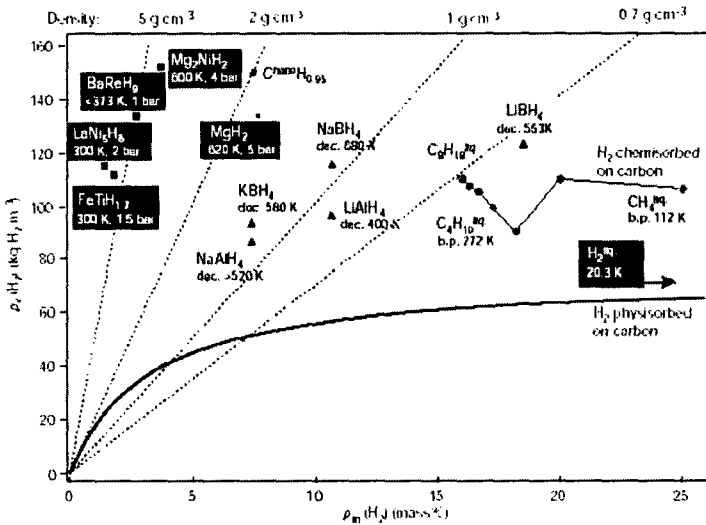
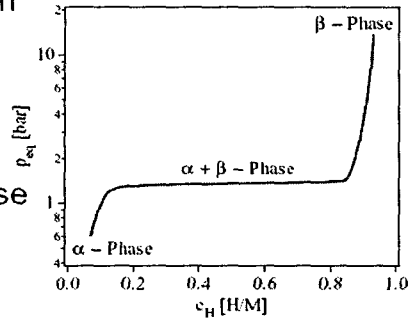
MH_x $x = \{1, 2, 3, \dots\}$
 $H \leftrightarrow H$



Absorption



Desorption



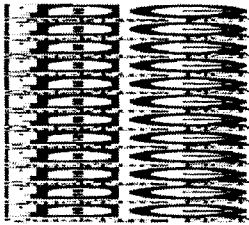
Low mass density = general weakness of all known MH working near RT

Intermetallic compounds & alloys can reach 9 wt.% but are not reversible within the required Ts

Stored hydrogen per mass and volume. Comparison of metal hydrides, carbon nanotubes, petrol and other hydrocarbons.

Hydrogen Storage In MH

■ Metal hydride storage



- ★ safety and long term stability
- ★ highest capacity by volume
- ★ high capacity by weight for Mg
- ★ free geometry

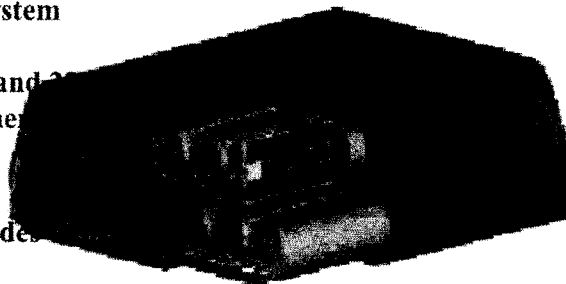
Problem: Storage & Application

- ⚡ high temperature of operation → 300°C
- ⚡ sluggish → refueling: ≈ several hours

Challenges in development of the storage, transport and distribution infrastructures

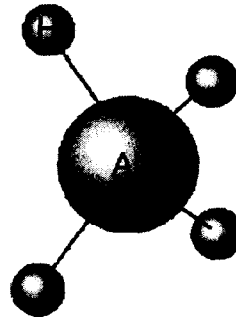
Metal Hydride Storage

- Current metal hydride system
= 1.5 – 5 wt.% H₂
√ Operate @ 300-400 °C and 20 bar
√ Primary challenge is the thermal management
- Low – temperature hydride storage development
√ Goal: 5.5 wt %H₂ @ <100 °C



Complex Hydride

- Complex hydrides consist of a $H=M$ complex with additional bonding element(s)
- hydrogen complexes include:
 - $(AlH_4)^-$ (alanates)
 - $(BH_4)^-$
 - with Group VIII elements
- features:
 - ionic, covalent, metallic bonding
 - can have lower formation energy
 - can have high H/M
- 173 complex hydrides listed on hydpark.ca.sandia.gov



Irreversible Chemical Hydrides

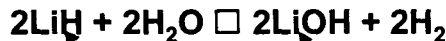
Three Approaches



20 - 35% sol.
Stabilized with
1-3% NaOH

Catalyst

Borax in NaOH



Light mineral oil slurry,
proprietary stabilizers

Paste
byproduct

- Hydrogen capacity is high at around 10 wt% hydrogen.
- Dehydrogenation kinetics are fast.
- Reactions are irreversible on-board vehicle.



Polyethylene-coated pellets,
mechanically cut to expose Na

Regeneration costs are
a major issue

수소연료전지 스쿠터 시운전 성공

수소에너지사업단

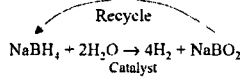
국내 기술진이 친환경 미래 에너지의 하나로 각광받는 수소연료전지를 장착한 스쿠터(사진)의 시험 운전이 성공했다.

고효율수소에너지제조저장이용기술개발사업단(단장 김종원)은 18일



한국과학기술연구원(원장 김유승), 삼성엔지니어링(대표 정연주)과 함께 촉매반응을 통해 수소 기체를 발생시키는 방식의 연료전지를 스쿠터에 장착, 1회 연료주입량인 6리터의 화학수소화물(NaBH₄) 수용액으로 140km 이상 주행할 수 있었다고 밝혔다.

이번에 개발한 전지는 기존 350기압 저장법보다 1.5~2배 가량 수소 저장밀도를 높일 수 있다.



Other complex hydrides: NaAlH₄ [7 wt%, 2 stage]
Beryllium hydrides Li₂BeH₄, 8 wt% [Toxicity issues]

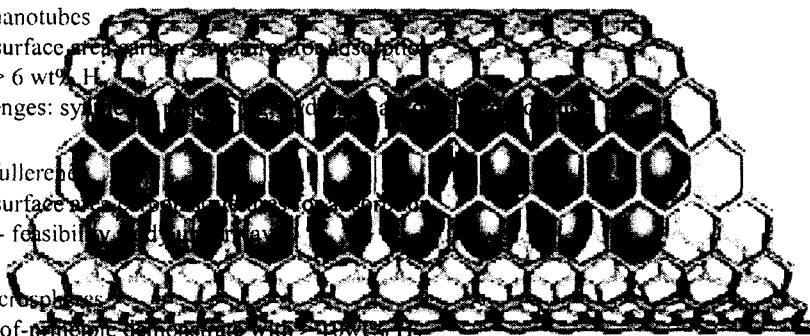
Complex Hydrides

- Reversibility
 - role of catalyst or dopant
- Thermodynamics
 - pressure, temperature
- Kinetics
 - long-range transport of heavy species
- Cyclic stability
- Synthesis
- Compatibility/safety

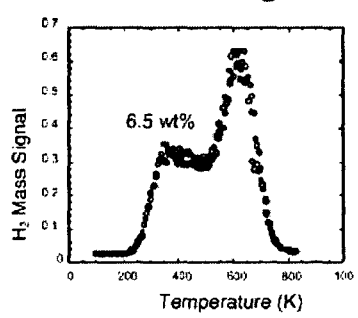
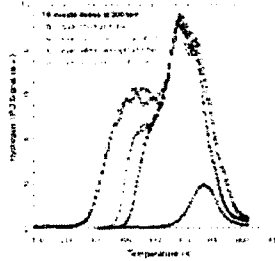
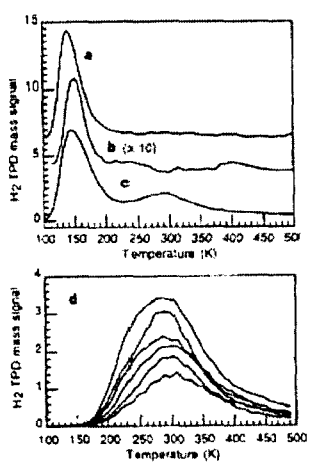
Examples	Theoretical reversible capacity/ wt%
Na(AlH ₄)	5.6
Li(AlH ₄)	7.9
Mg(AlH ₄) ₂	7.0
Ti(AlH ₄) ₄	8.1
Fe(BH ₄) ₂	9.4
Na(BH ₄)	7.9
Ca(BH ₄) ₂	8.6

Advanced Solid-State Storage

- Carbon nanotubes
 - ✓ High surface area
 - ✓ Goal > 6 wt% H₂
 - ✓ Challenges: synthesis, processing
- Carbon fullerenes
 - ✓ High surface area
 - ✓ Status- feasibility
- Glass microspheres
 - ✓ Proof-of-principle demonstration
 - ✓ Potential for low cost, high-capacity conformable storage
 - ✓ Challenges: synthesis, processing, thermal/pressure management of adsorption/desorption

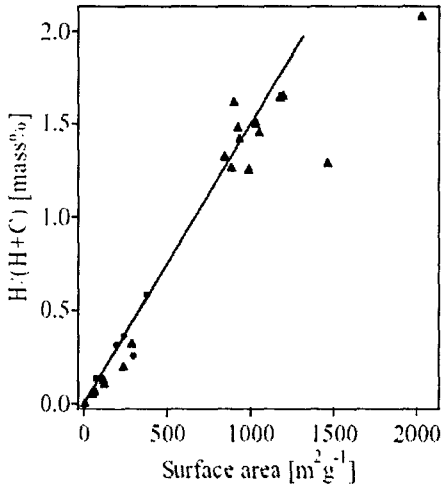


Hydrogen in SWCNT



Hydrogen TPD spectrum of a degassed sample. room temperature H₂ exposure at 500 torr. The adsorbed hydrogen corresponds to 6.5 wt%. Hydrogen TPD data from an SWCNT sample that was exposed to hydrogen at 300 Torr for 10 minutes followed by a variation in post-dosing conditions.

HYDROGEN STORAGE IN CARBON NANOTUBES



Ref.: M.G. Nijkamp, J.E.M.J. Raaymakers, A.J. van Dillen, K.P. de Jong, Appl. Phys. A 72 (2001), pp. 619–623

Hydrogen gas adsorption at
77 K
electrochemical capacity at
293 K

1.5 mass% / 1000 m²g⁻¹
max. 2 mass%

Hydrogen gas adsorption at
296 K and 125 bar:
max. adsorption 1.5 mass%

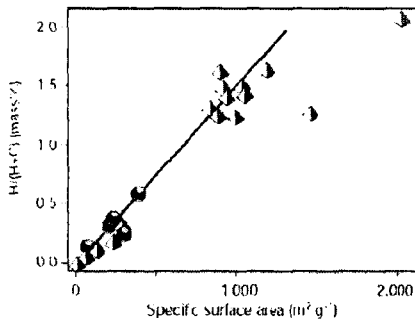
Ref.: R. Ströbel et al., Journal of Power Sources
84 (1999), pp. 221-224

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Carbon materials - an alternative to high density storage...

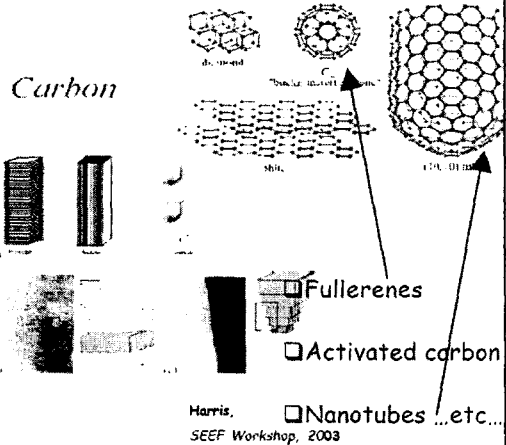
Hydrogen absorbs at solid surfaces depending on the applied pressure & temperature

Reversibly stored amount of hydrogen
on various carbon materials vs. the
specific surface area of the samples



● nano-tube samples (first fit line indicated)
▲ other nano-structure carbon samples

Schlapbach & Züttel, Nature, 414, 353-358, 2001



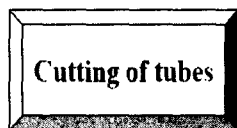
Nanostructured graphitic carbon at 77K amounts to 1.5% mass per 1,000m²/g surface area. Temperature dependent - at 77k one order of magnitude higher than at 300K.

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The key point of H₂ storage

block the hydrogen diffusion

A structural defect in the tube
A sharp bend of the tube

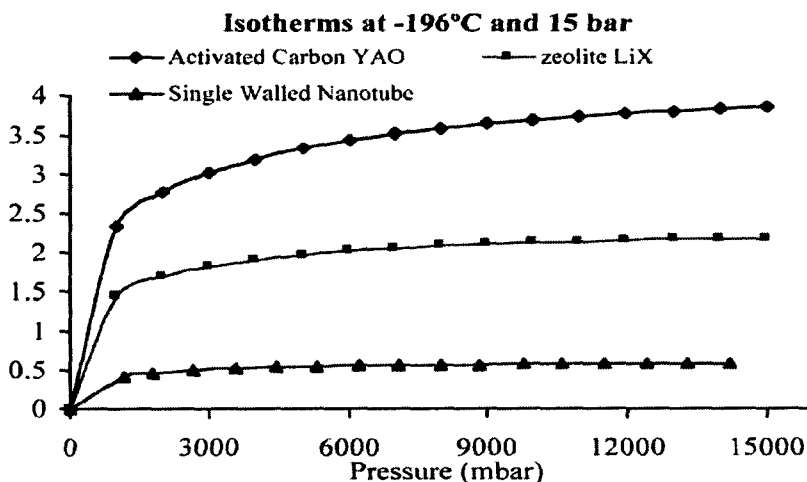


Sonication time
Sonication power
Acid concentration
Hydrodynamics



Amounts of metal
Metal particle sizes
Improvement of H₂ storage

IGA traces (PCT plots) of activated carbon, zeolite and SWNT material at -196°C up to 15 bar with 1 bar pressure steps.



Hydrogen Storage in Carbon

Material	Density wt%	Temp (K)	Pressure (MPa)	Reference	Year
GNFs (herring bone)	67.55	RT	11.35	Chambers	1998
GNFs (platelet)	53.08	RT	11.35	Chambers	1998
LI-MWNTs	2.0	~473-673	0.1	Chen	1999
K-MWNTs	1.4	< 313	0.1	Chen	1999
GNFs (tubular)	11.26	RT	11.35	Chambers	1998
CNFs	~10	RT	10.1	Fan	1999
LI/K-GNTs (SWNT)	~10	RT	8-12	Gupta	2000
GNFs	~10	RT	8-12	Gupta	2000
SWNTs (100% purity)	6-10	273	0.04	Dillon	1997
SWNTs (50% purity)	8.25	80	7.18	Yo	1999
CN nanobelts	9	573	0.1	Bal	2001
Nano graphite	7.4	RT	1	Orlino	2000
SWNTs (hi p + Ti alloy)	6-7	~300-700	0.07	Dillon	2000
GNFs	6.5	RT	~12	Browning	2000
CNFs	~5	RT	10.1	Cheng	2000
MWNTs	~5	RT	~10	Zhu	2000
SWNTs (hi p + Ti alloy)	3.5-4.5	~300-600	0.07	Dillon	1999
SWNTs (50% purity)	4.2	RT	10.1	Liu	1999
LI-MWNTs	~2.5	~473-673	0.1	Yang	2000
SWNT (50% purity)	~2	RT	0.04	Nutzenadol	1999
K-MWNTs	~1.8	< 313	0.1	Yang	2000
(9,9) array	1.8	77	1.0	Wang	1999
MWNTs	< 1	RT	0.04	Beguin	2000
CNF	0.1-0.7	RT	0.1-10.5	Poirier	2001
(9,9) array	0.6	RT	1.0	Wang	1999
SWNTs	~0.1	300-520	0.1	Hirschner	2000
Various	< 0.1	RT	3.5	Tibbets	2001
SWNT (+ Ti alloy)	0	RT	0.08	Hirschner	2001

> 10 wt%

< 1 wt%


Comparison of H₂ storage

Different types								
	Process	Energy involved in the process	Storage pressure	Temperature (°C)	Volumic density	mass ratio (%)	specific energy	Energy needed for H ₂ release
compressed	Multiple steps adiabatic compression - cooling	3.3 kWh/kg H ₂	200-350 to 800 bar	25 °C	max. 33 kg H ₂ /m ³	1.1 to 2.6 (200 bar)	0.45 to 5 kWh/kg	0
Underground storage	Aquifers Salt caverns		80 - 160 bar			11.5-13 (700-800 bar)		
liquefied	Liquid N ₂ cooling - Multiple steps adiabatic compression	10 kWh/kg H ₂	No data	-253 °C	71 kg H ₂ /m ³	26	14 kWh/kg	0 1 to 3 % loss per day
NaBH ₄ H ₂ storage on demand	NaBH ₄ + 2 H ₂ O + catalyst → NaBO ₂ + 4 H ₂	No data	No data	No data	No data	No data	No data	0
Metal hydrides	chemisorption / desorption	No data	2 to 10 bar	0-100 °C	max. 150 kg H ₂ /m ³	2	0.8 to 2.3 kWh/kg	No data
Active carbons	physisorption / desorption	No data	250 bar	25 °C	18 kg H ₂ /m ³	No data	2.2 kWh/kg	No data
Carbon nanofibres & nanotubes	physisorption / desorption	No data	125 bar	25 °C	No data	1.5	1.7 to 3 kWh/kg	No data
fullerenes	chemisorption / desorption	100 kJ/mol (14 kWh/kg of H ₂)	27 bar	100-200 °C	No data	6-7.7	2.5 kWh/kg	160 kJ/mol (22 kWh/kg H ₂)

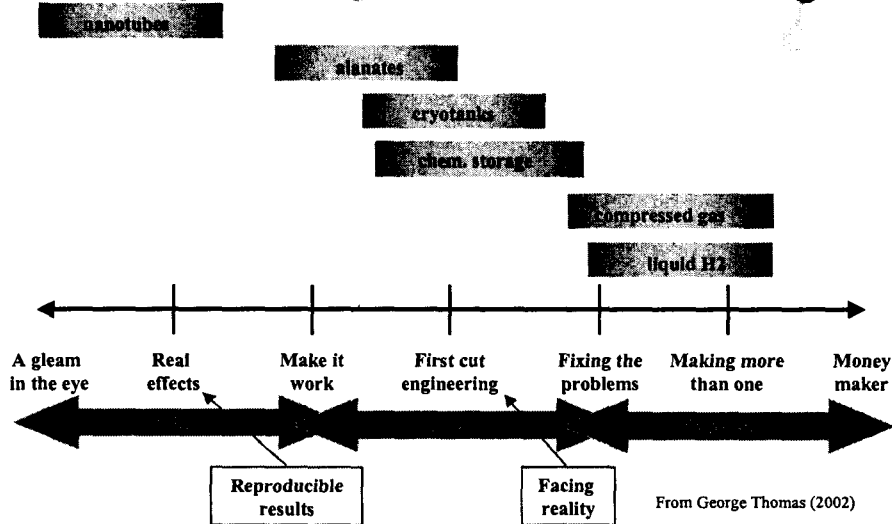
Maximum storage densities

Energy Density MJ/liter

• High pressure gas				
• ambient temperature	3600 psi:	2.0	5000 psi:	2.75
• cryogenic system	150 K:	3.5	20 K:	8.4
• Liquid hydrogen		8.4		
• Reversible storage media				
• carbon structures				
• nanotubes		?		
• fullerenes		?		
• hydrides				
• intermetallics		10.8 - 12.0		
• alanates		8.25		
• composite materials		?		
• Chemical methods	<u>Eff.</u>	<u>gasoline</u>	<u>methanol</u>	
• liquid fuel + reformer		50%:	6.6	5.9
•		75%:	9.9	8.9
• off-board reprocessing		?		

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*The most promising technologies
are the farthest from commercialization*

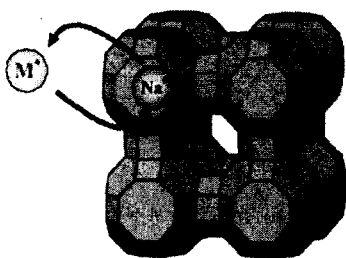


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Advanced storage approaches identified

1. Crystalline Nanoporous Materials
2. Polymer Microspheres
Self-Assembled Nanocomposites
3. Advanced Hydrides
4. Metals – Organic
5. BN Nanotubes
Hydrogenated Amorphous Carbon
6. Mesoporous materials
7. Bulk Amorphous Materials (BAMs)
8. Iron Hydrolysis
9. Nanosize powders
10. Metallic Hydrogen
Hydride Alcoholysis

Hydrogen Uptake in Zeolite



Zeolite A

- Low cost, chem.&therm. Robust
- Good structural reproducibility
- Environmentally friendly & safe

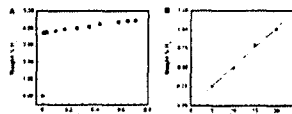
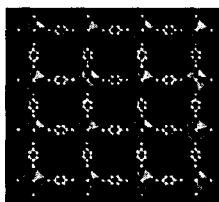
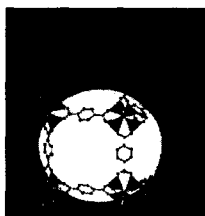
Material	H ₂ uptake (wt.%)		
	-196°C	RT	27°C
NaA	1.54	0.28	0.30
CdA	1.14	0.25	0.30
MgA	1.19	-	-
NaC-RHO	0.00	0.18	0.20
CdRHO	0.08	0.19	0.25
LiX	2.15	-	-
NaX	1.79	-	0.25
CdX	1.42	-	-
MgX	1.61	-	0.28
CdY	1.47	-	-
MgY	1.74	-	-

15bar H₂

Mesoporous Metal Organic (MOF-5)



Single-crystal X-ray structures of MOF-5(A), IRMOF-6B, and IRMOF-4(C). Zn (blue) and O (red) spheres; C (black) spheres

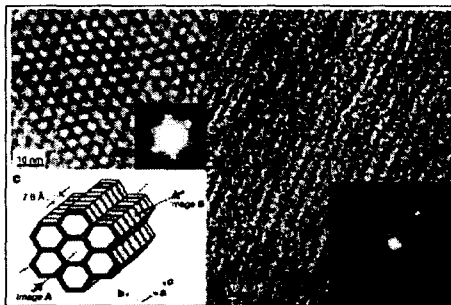


Hydrogen gas adsorption for MOF-5 at 75 K (A) and at RT and 20 bar (B)

Hydrogen gas adsorption for MOF-5 at 75 K and B) 295 K

- Chemical formula $Zn_4O(BDC)_3(DMF)_2$ (C_6H_5Cl)
 - BDC = 1,4 - benzenedicarboxylate
 - DMF = dimethylformamide
- ZnO_4 tetrahedral clusters linked together by $C_6H_4-C-O_2$ "struts"
- Cubic crystal structure
- 1.294 nm spacing between centers of adjacent clusters
- What are the hydrogen storage characteristics of this material?

Mesoporous Organosilica Material



benzene-silica hybrid material
Hydrogen storage behavior?

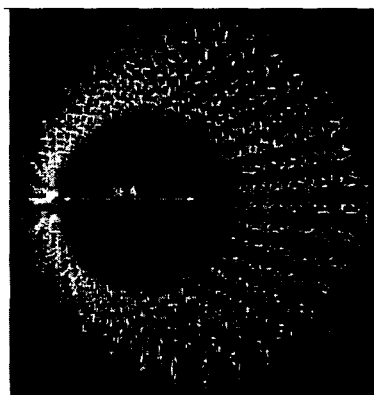


Figure 4 Model showing the pore surface of mesoporous benzene-silica. Benzene rings are aligned in a circle around the pore, flanked at both sides by silicate chains. The silicate is terminated by silanol (Si-OH) at the surface. Hydrophobic benzene layers and hydrophilic silicate layers array alternately at an interval of 7.6 Å along the channel direction. Silicon, orange; oxygen, red; carbon, white; hydrogen, yellow

Boron Nitride Nanotube

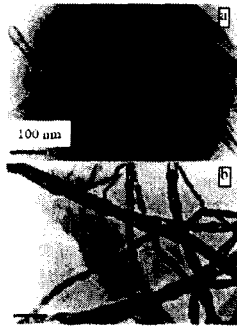


Figure 1. The multiple layers of BN nanotubes: (a) multi-wall nanotubes, and (b) bamboo-like nanotubes. Scale bar = 100 nm.

Multiwall : 1.8 wt%
Bamboo-like : 2.6 wt%

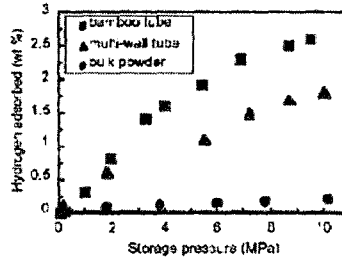
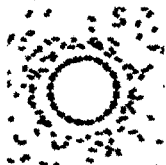


Figure 2. The hydrogen adsorption is a function of pressure in multi-wall BN nanotubes and bamboo-like nanotubes at 10 MPa is 1.8 and 2.6 wt%, respectively, in sharp contrast to the 0.2 wt% in bulk BN powder. The values reported here have an error of ± 0.3 wt%.

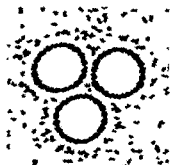
Hydrogen Storage in Inorganic Nanostructured Materials at KBSI

Hydrogen Storage in NT

Single tube, T=265



Three tubes, T=265



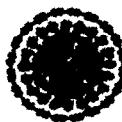
Three tubes, T=77



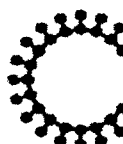
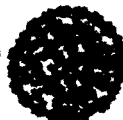
Physisorption: Results suggest that tubes should be kept mechanically separated.



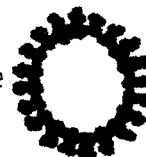
After simulation



After simulation



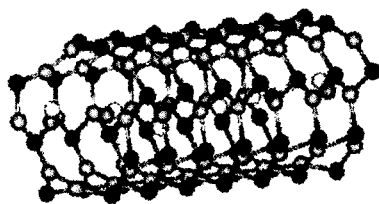
After simulation



Chemisorption inside and outside tube: inside is not stable but outside is stable

H₂ Storage in LiAlO₂ Nanotubes

α -LiAlO₂



Armchair

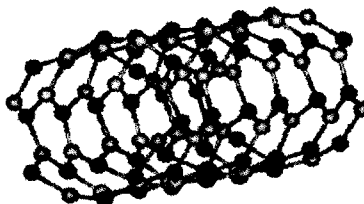
Surface Area : 817 m²/g



ZigZag

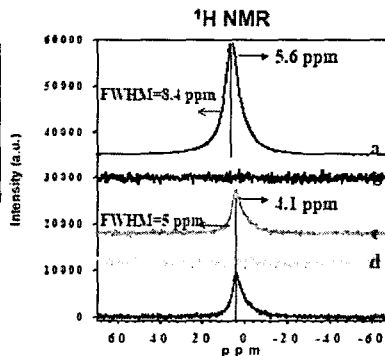
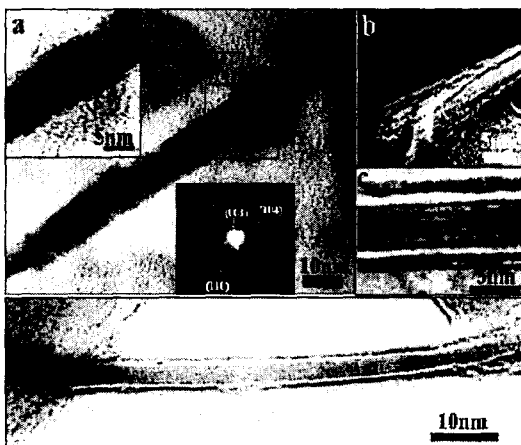
Surface Area : 1027 m²/g

β -LiAlO₂



Surface Area : 969 m²/g

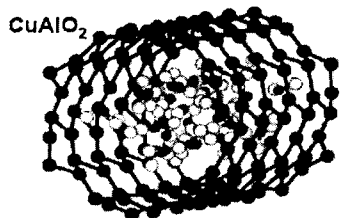
H₂ storage in LiAlO₂ Nanotube



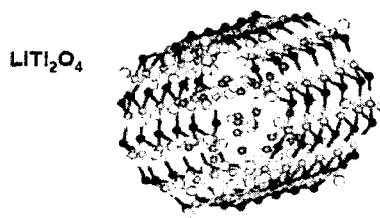
- (a) P-ANT-Li before evacuation
- (b) Evacuation at RT 54h
- (c) H₂ adsorption at RT, 2.7atm 45h
- (d) Re-evacuation at RT 10min
- (e) H₂ re-adsorption at RT, 2.7atm 45h

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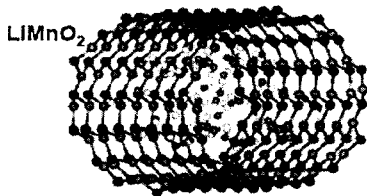
H₂ uptake in M-oxide Nanotubes



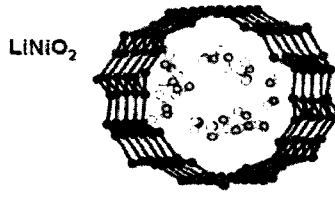
Surface Area : 1128 m²/g



Surface Area : 1860 m²/g



Surface Area : 2949 m²/g

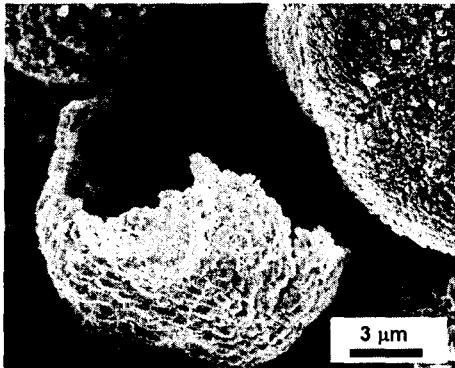


Surface Area : 1322 m²/g

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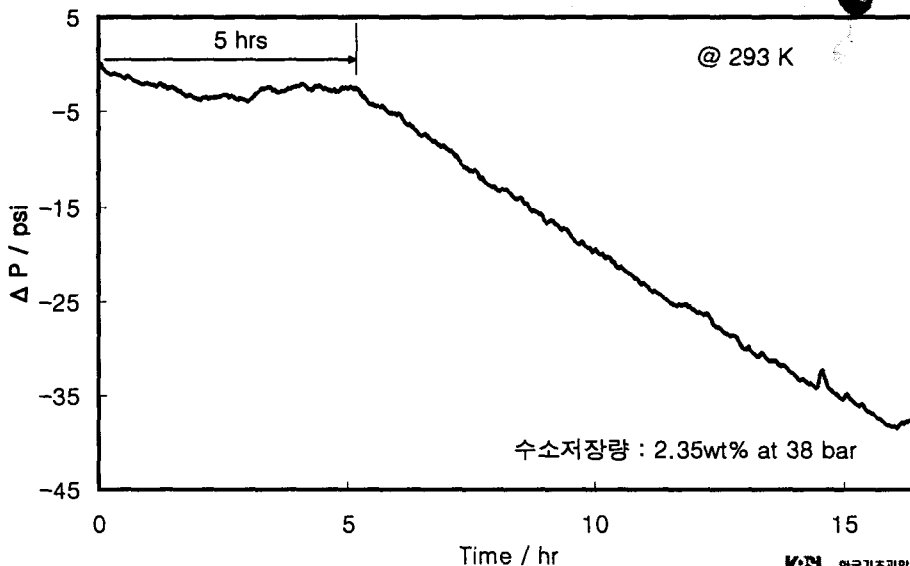
$\text{Cu}_2\text{Cl}(\text{OH})_3$ microspheres

Biomimetic control을 통한 $\text{Cu}_2\text{Cl}(\text{OH})_3$ aggregate microsphere



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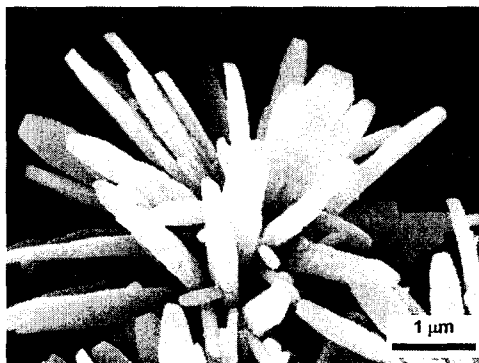
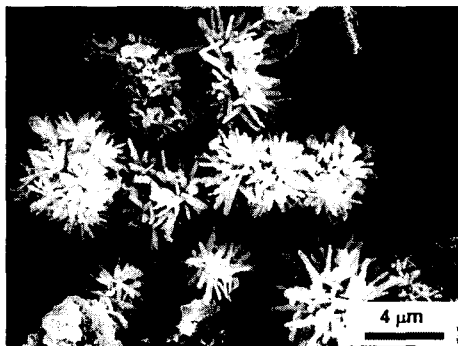
H_2 Storage in calcinated $\text{Cu}_2\text{Cl}(\text{OH})_3$ microspheres



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CuAlO₂ nanoflower

계면활성제의 상평형도를 이용한 morphology 제어와 이것을 Template로 고상법을 이용하여 CuAlO₂ 나노구조체 합성



H₂ Storage : 0.66 wt% @RT, 45 bar

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Candidate for H₂ Uptake Materials LiMnO₂ Nanomaterials



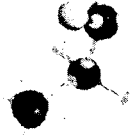
SEM Images



TEM Images

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Summary



- Hydrogen storage, even though still at its infancy, appears as a possible attractive alternative.
- Improved safety and energy density

A breakthrough in Hydrogen storage technology could facilitate the introduction of H economy society.